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(72) Inventor: JAN VERLOOP

(54) METHOD FOR THE PREPARATION OF A GAS MIXTURE HAVING A DESIRED HS/CO2 RATIO

SHELL TIONALE RESEARCH MAATSCHAP- liquid absorbent. PIJ B.V., a company organised under the and the method by which it is to be per-elemental sulphur, which is separated off. formed, to be particularly described in and by the following statement:-

(hereinafter referred to as the starting gas) in order to prepare a gas mixture having an H₂S/CO₂ ratio suitable for use in a Claus suitable catalyst. process (hereinafter referred to as the de-

sired gas).

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the said gas mixtures are discharged to the for the operation of a Claus process: atmosphere.

generally has to be removed are gases of the gas mixture consists of HaS (and the obtained by complete or partial combustion rest is virtually entirely CO2), the first stage or gasification of oil and coal, refinery of the Claus reaction can be carried out by

water gas, propane and propylene.

character. In a large number of cases the gas suitable catalyst. mixtures to be purified will also contain in the liquid absorbent together with the tially H2S and CO2, consists of between 15-H.S. The H.S and CO₂ will be removed from 20 mol. % of H₂S on the one hand and the said gas mixtures at the pressure of the approximately of 40 mol.% of H₂S on the gas mixture concerned, i.e., in many cases other, the above-mentioned method en-at elevated pressure. By heating the loaded counters difficulties and the Claus process liquid absorbent, for example with steam, a has to be carried out by separating one-third

INTERNA- these substances were present in the loaded

This H₂S and CO₂-containing gas mixture laws of The Netherlands, of 30 Carel van cannot be discharged to the atmosphere be-Bylandtlaan, The Hague, The Netherlands, fore at least most of the H2S has been redo hereby declare the invention, for which moved therefrom. The H2S is very suitably we pray that a patent may be granted to us, removed from this gas by converting it into

The conversion of H2S into elemental sulphur is generally carried out in the art by The invention relates to a method for in-creasing the H₂S/CO₂ ratio in a gas mixture the H₂S is oxidized to SO₂, and sulphur and water are formed by reaction of H2S with SO₂, with or without the assistance of a

For the operation of a Clause process with a gas mixture comprising substantially H-S In many cases it is necessary to remove and CO2, the method to be followed de-H.S from gas mixtures, for example in order pends on the ratio of H.S and CO2 in this gas to render these gas mixtures suitable for mixture, or in other words, on the molar catalytic conversions using sulphur-sensitive percentage of H.S based on the total amount catalysts, or in order to reduce environmen- of H2S and CO2 present. Three different H2S tal pollution if before or after combustion concentration ranges can be distinguished

Examples of gas mixtures from which H.S 1) If more than approximately 40 mol. % gases, town gas, natural gas, coke-oven gas, combusting the gas mixture with a quantity of air which is sufficient to convert one-third In many cases the Hs will be removed part of the H2S into SO2. In one or more from the said gas mixtures using liquid subsequent reactors, sulphur and water are absorbents, which will often have a basic then formed, preferably in the presence of a

CO2, at least some of which will be absorbed 2) If the gas mixture comprising substangas mixture is obtained consisting substanpart of the gas and combusting the H₂S tially of H₂S and CO₂ in the ratio in which therein to SO₂, and subsequently mixing the 90

(19)

resultant SO-containing gas with the rest of the HS-containing gas, after which the Claus reaction can be further carried out at elevated temperature and preferably in the presence of a catalyst.

3) If less than 15–20% by volume of the gas mixture consists of H-S (and the rest virtually entirely of CO₂), a Claus process cannot be carried out in the above-mentioned manner. The combustion in the gas mixture has to be additionally supported. This may be effected, for example, by recycling the sulphur formed in the Clause process to the H₂S and CO₂-containing gas, or by oxidation of the latter gas with pure oxygen instead of air, or by injecting a fuel (for example a hydrocarbon) into the gas before it is combusted in the thermal stage of the Claus process. These processes are generally expensive and not very attractive technically.

Another possibility to raise the H-S/CO₂ ratio in the mixture consists in selectively absorbing H₂S from the gas mixture in an appropriate absorbent, and subsequently liberating the H₂S (mixed with the absorbed CO₂) from the loaded absorbent in a regeneration stage. In case the H₂S concentration in the gas mixture is low, an excessive amount of CO₂ is still absorbed in the absorbent during the selective absorption, when using this method. For that reason it sometimes happens that no gas mixture having the desired composition is obtained after liberation of the absorbed gas from the absorbent loaded in this way.

There is therefore a need for a process whereby the quantity of H₂S is a gas mixture, in particular in a gas mixture otherwise consisting substantially of CO₂, can be increased according to desire. It will be endeavoured to control the H₂S content in such a way that either a Claus process can be carried out according to the system described first (addition of air to the entire mixture) or according to the system described second (separation of one-third part of the gas mixture and complete combustion of the H₂S present therein, followed by combination with the rest of the gas mixture).

Because there are in general no great differences between the costs of investment and operation of the embodiments of the Claus process mentioned in 1) and 2), a process whereby the quantity of H₂S is a gas mixture can be increased according to desire will be of importance mainly to increase H₂S concentrations of below 15-20% to 20% or more. At the same time ti will be endeavoured not to increase the H₂S concentration much higher than is needed in order to be able to carry out the method of the Claus process described in 2), because higher concentrations do not offer any important advantages in the Claus process,

whereas they do require more absorption and regeneration capacity and energy.

The invention provides a method for the preparation of a gas mixture having a desired H₂S/CO₂ ratio, independent of the originally present H₂S/CO₂ ratio in the starting gas, as a result of which an optimum use of Claus plants and absorbent/regeneration equipment can be obtained.

According to the invention there is provided a method for increasing the HiS/CO2 ratio in a gas mixture (hereinafter referred to as the starting gas) in order to prepare a gas mixture having an H₂/CO₂ ratio suitable for use in a Claus process (hereinafter referred to as the desired gas), which process is characterized in that H₂S and CO₂ of the starting gas are absorbed in an aqueous solution of an amine (further to be called an amine liquid absorbent) in an absorption 85 column, in that the absorbed gas is liberated from the resultant loaded amine liquid absorbent in a regeneration column, in that a portion of the gas becoming available from the regeneration column is treated with amine liquid absorbent in an absorption column under such conditions that HS is absorbed selectively relative to CO2 in the amine liquid absorbent and the loaded amine liquid absorbent obtained in this 95 manner is introduced into the said regeneration column, and in that the magnitude of the said portion of the gas becoming available from the regeneration column is so selected that this gas is the desired gas, and 100 the remainder of the gas becoming available from the regeneration column (further referred to as the resultant gas) is removed.

The method according to the invention may be carried out in a number of different ways, depending on the composition of the starting gas, and on the H₂S/CO₂ ratio in the desired gas.

In addition to Hs and CO₂, the starting gas will generally contain other components, and in a first absorption step the starting gas will be treated with an amine liquid absorbent in which Hs and CO₂ are absorbed, while the other gases of the starting gas are not or only slightly absorbed. 115 Such a treatment yields an amine liquid absorbent loaded with Hs and CO₂, the ratio of these gases in the amine liquid absorbent remaining the same or substantially the same as that in the starting gas.

In a number of cases it will be possible to prepare the desired gas according to the invention by introducing the above-mentioned H₂S and CO₂-loaded amine liquid absorbent into a regeneration column and to treat a proportion of the gas liberated from the regeneration column, which gas comprises substantially H₂S and CO₂, in an absorption column under such conditions that H₂S is absorbed in the amine liquid 136.

an absorption column is hereinafter also re- berated from the regeneration column. ferred to as the selective absorption column. The H₂S-loaded amine liquid absorbent becoming available from the selective absorption by means of this method, because the tion column is subsequently supplied to the ratio of the quantities of H.S and CO2 in the regeneration column simultaneously with starting gas is too low. Also, in certain cases the aforementioned loaded amine liquid it is possible to obtain a gas having the deabsorbent (which will generally be loaded sired composition, but to that end so large a almost exclusively with H₂S and CO₂). Both proportion of the desired gas becoming streams can be introduced into the regener- available from the regeneration column has ation column separately, but it is preferred to be passed to the selective absorption colto mix the streams before supplying them to umn that the method becomes technically the regeneration column. In the regeneration column the CO₂ and H₂S are completely In such cases it is possible to the amine liquid absorbent by heating, very suitably by heating with steam.

generation column must have the desired components as well, to be liberated from the composition, and this composition will be loaded amine liquid absorbent in a regenerdependent on the quantity of H.S which is ation column. Subsequently, the H2 S in the supplied to the regeneration column in the gas set free from the latter regeneration form of H₂S-loaded amine liquid absorbent column (which gas in this embodiment is originating from the selective absorption considered as starting gas), is selectively column. The latter quantity of H₂S is dependent on the quantity of H₂S supplied to the absorption column, and the resultant H₂Sselective absorption column, because that loaded amine liquid absorbent is supplied to H.S is absorbed substantially completely in a regeneration column. the amine liquid absorbent in the said

absorption column. liquid absorbent in the selective absorption also supplied to the regeneration column. column will be far higher than in the aforebe supplied to the regeneration column; as a result the H₂S/CO₂ ratio in the regeneration column and therefore in the gas mixture beof H.S supplied from this gas mixture to the selective absorption column; that quantity of HS is determined by the portion of the

selective absorption column, it is therefore gas to be treated. possible according to the invention to adjust (generally loaded exclusively with H₂S and umn either together or separately. In the CO₂) to be supplied to the regeneration collatter case it is advantageous to introduce

absorbent selectively relative to CO2. Such umn will invariably be present in the gas li-

In a number of cases it will not be possible to obtain any gas having a desired composi-

In such cases it is possible to carry out the or substantially completely removed from method according to the invention in another manner. It is then very suitable for the H₂S and CO₂ absorbed in the amine li-The gas mixture liberated from the re-quid absorbent from gases containing other

A portion of the desired gas becoming available from the said regeneration column Because not more than a portion at most is treated with amine liquid absorbent in a of the CO2 is absorbed in the amine liquid selective absorption column, and the HaSabsorbent in the selective absorption colloaded amine liquid absorbent obtained umn, the Hs/CO2 ratio in the loaded amine from the said selective absorption column is

It is possible to use two absorption colmentioned loaded amine liquid absorbent to umns in which Hs is selectively absorbed, namely one for the treatment of the starting gas and one for a portion of the desired gas. If desired, the loaded amine liquid absorcoming available therefrom is higher than in bent obtained by selective absorption of the the afore-mentioned loaded amine liquid starting gas may be introduced into the absorbent to be supplied to the regeneration selective absorption column for the desired column. The H₂S/CO₂ ratio in the gas mix- gas, where it can serve as absorbent for the ture liberated from the regeneration column said desired gas. In the selective absorption is consequently determined by the quantity column for the desired gas completely regenerated amine liquid absorbent may optionally be introduced in addition.

It will be clear from the above that by the gas mixture liberated from the regeneration term "amine liquid absorbent" is meant not column which is supplied to that absorption only an aqueous solution of an amine containing substantially no gas (i.e., completely By controlling the magnitude of the por- regenerated), but also such a solution partly tion of the gas mixture liberated from the loaded with gas, provided that this load is regeneration column and supplied to the such that H.S can still be absorbed from the

It is generally preferred to use a single as desired the H.S/CO2 ratio in the gas mix- selective absorption column in which both ture liberated from the regeneration col- the above-mentioned gases, i.e., starting gas umn; this ratio is naturally limited in that and desired gas, are treated with amine li-the quantity of CO₂ present in the afore- quid absorbent. These gas streams may be mentioned loaded amine liquid absorbent introduced into the selective absorption col-

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the desired gas at a point below the point of introduction of the starting gas.

It may occur that fluctations in the H2S/ CO2 ratio occur with time in the starting gas containing H2S and CO2 in a ratio lower than that desired. In order to keep the H.S/CO2 ratio constant or substantially constant in the gas mixture to be obtained according to the method of the invention, the quantity of gas mixture to be supplied to the selective absorption column will have to be varied (and if desired also the quantity of amine liquid absorbent to be supplied to the said column). It may even occur that the H-S/ CO₂ ratio in the starting gas is the same or higher than the desired ratio, so that no further H2S enrichment of the gas mixture obtained from the regeneration column is necessary. In that case a method according to the invention is not necessary and the introduction of the desired gas into the selective absorption column can be terminated; if the desired gas is the only gas being supplied to the selective absorption column, the said column can be shut down completely.

As an illustration of a process in which the quantity of the gas becoming available and the H2S/CO2 ratio therein may fluctuate, mention may be made of the gasification of coal. First, most of the H-S and CO2 will be removed by non-selective absorption from a gas obtained in this process, and subsequently H₂S and CO₂ will be liberated from the thus loaded amine liquid absorbent. H.S. is removed by selective absorption in a selective absorption column from the resultant gas mixture consisting substantially of H₂S and CO₂, and by regeneration of the resultant H2S-loaded liquid amine absorbent a gas is obtained which under normal process conditions is suitable for a Claus process. If in the case of reduction of the supply of gas obtained by coal gasification and/or decrease of the H₂S content thereof no gas is obtained which is suitable for the Claus process, it is possible according to the method of the invention to introduce into the said selective absorption column a portion of the gas becoming available from the regeneration column.

The amine liquid absorbent used may be an aqueous solution of primary, secondary and tertiary amines or mixtures thereof. As an example mention may be made of ethanol amine; preference is given to polyalkanol amines, in particular di-isopropanol amine and methyl diethanol amine.

If desired, the aqueous solution of an amine may also contain non-basic compounds capable of physically absorbing H.S and CO₂. Examples of this type of compounds are N-methyl pyrrolidone, methanol be freed of HS is supplied via a line 1 to an and cyclotetramentylene sulphones, in particular sulfolane.

absorption column are so controlled that H₂S is selectively absorbed from the gas mixture, which otherwise consists largely of CO2. The process is very suitably operated countercurrently in a tray column, the gas mixture to be absorbed being introduced at the bottom of the column. The absorption column preferably contains fewer than 20 trays, and the gases are passed through the column at a considerable rate, preferably at a rate of at least 1 m/sec., based on what is known as the "active" or "aerated" surface of the contact tray. If desired, a packed bed absorption column may also be used, having a packed height of preferably less than 10m.

Preferably, the gas mixture to be absorbed is contacted in each selective absorption column with the amine liquid absorbent at approximately atmospheric pressure, although higher and lower pressures are by no means excluded. The temperature of the amine liquid absorbent in the selective absorption column may vary within wide limits; temperatures below 55°C, in particular from 15 to 50°C, are very suitable, although higher temperatures are by no means excluded.

The resultant gas mixture containing H2S and CO2 in the desired ratio (i.e., for use in a Claus process containing either 20 mol. % or slightly more of H-S or 40 mol% or slightly more of H₂S, as explained above) is very suitably subjected to a Claus reaction, in which sulphur is formed. The off-gases from the Claus process, which still contain some H₂S, can be discharged to the atmosphere after combustion, or their content of sulphur compounds can be further reduced to a considerable extent by reduction and selective absorption in a liquid absorbent of the gas obtained after reduction, for example as described in the published Netherlands patent application 7010604.

The gas becoming available from the selective absorption column, which gas comprises substantially CO2 but will generally still contain a minor quantity of H.S, can also be discharged to the atmosphere after combustion. Combustion and/or discharge thereof may of course be effected together with other sulphur-containing gases, such as the abovementioned Claus off-gases. In case two absorption columns are present, the gas becoming available from the selective absorption column may also (if desired partially) be recycled to the first absorption column.

The invention will now be illustrated with reference to Figs. 1 and 2, each of which represents an embodiment of the invention.

In Fig. 1 a CO2-containing gas which must absorption column 2. The purified gas is discharged via a line 3. The H.S and CO2-The structure of and the conditions in the loaded amine liquid absorbent is discharged

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via a line 4 and passed to a regeneration column 5. The gas becoming available from the regeneration column 5 via line 6 (the desired gas) is divided into two portions. One portion is discharged via a line 7, for example to a Claus plant 8, the discharge gas of which is discharged to the atmosphere via a line 9, an incinerator 10 and a line 11. The second portion of the gas becoming available from the regeneration column 5 is supplied via a line 12 to a selective absorption column 13 according to the invention. The gas liberated from the said absorption column is either supplied via a line 14 and the line 9 to the incinerator 10, or it is recycled via a line 19 to absorption column 2. The H₂S-loaded amine liquid absorbent obtained from the absorption column 13 is supplied to the line 4 via a line 15. At the point where the line 6 branches into the lines 7 and 12 there is a mechanism by means of which the distribution of the gas stream from the line 6 between the lines 7 and 12 can be controlled.

The amine liquid absorbent becoming available from the regeneration column via a line 16, which absorbent is free or substantially free of CO2 and H2S, is supplied via lines 17 and 18 to the absorption columns 2

and 13, respectively.

In Fig. 2 a gas comprising substantially H₂S and CO₂ is supplied via a line 101 to a selective absorption column 102. The gas issuing from the said absorption column is discharged via a line 103, and, because it still contains only very little H.S, it may, if desired, be discharged to the atmosphere after combustion. The H₂S and CO₂-loaded amine liquid absorbent is passed via a line 104 to a regeneration column 105. The gas becoming available from the said column via a line 106 (the desired gas) is divided into two portions. One portion is discharged via a line 107, for example to a Claus plant (now shown). The second portion of the gas becoming available from the regeneration column 105 is supplied via a line 112 to the selective absorption column 102. The amine liquid absorbent becoming available from the regeneration column 105, which absorbent is free or substantially free of H₂S and CO₂, is passed via a line 117 to the absorption column 102. At the point where the line 106 branches into the lines 107 and 112 there is a mechanism by means of which the distribution of the gas stream between the lines 107 and 112 can be controlled.

EXAMPLE I

The reference numerals refer to Fig. 1.

A gas stream of 80,000 Nm³/h, comprising substantially H₂ and CO and having an H₂S content of 0.3 mol.% and a CO₂ content of absorption column 2, which contains 15 con- by weight solution of methyl diethanol tact trays. Via line 17 a 30% by weight solu- amine in water, is introduced via the line 130

tion of methyl diethanol amine in water is supplied at a rate of 140 m³/h, and the gas mixture is treated therewith at a temperature of 45°C and a pressure of 20 bar abs. The purified gas escaping via the line 3 con- 10 tains 3.3% by volume of CO₂ and 10 ppm by volume of Has. The resultant Has and CO₂loaded absorbent, in which the H2S/CO2 molar ratio is 0.078, is supplied via the line 4 to the regeneration column 5. This regener- 75 ation column, which contains 14 trays, is heated with steam to a bottom temperature of 110°C, the bottom pressure being 1.4 bar abs. Via the line 6 a gas stream of 4150 Nm³/ h leaves the regeneration column, of which 80 gas stream 2950 Nm³/h is supplied via the line 12 to the absorption column 13, which contains 9 contact trays. A 30% by weight solution of methyl diethanol amine in water is supplied (45 m³/h) to this column via the 85 line 18, and the gas mixture is treated therewith at a temperature of 45°C and a pressure of 1.1 bar abs. The gas velocity is 1.5 m/sec. In the loaded absorbent discharged via the line 15, the H₂S/CO₂ molar ratio is 3.17, and 90 the said loaded absorbent is mixed with the absorbent present in the line 4, which absorbent contains H2S and CO2 in a ratio lower than the desired ratio. Via the line 14 a gas containing 90 mol.% of CO₂, 10 mol.% of H₂O and 1500 ppm by volume of H₂S is discharged from the absorption column 13.

As a result of the method according to the invention, the H2S/CO2 ration, which was 0.078 in the absorbent in the line 4, has been 100 increased to 0.28 in the gas mixture becoming available from the regeneration column via the line 6. The said gas mixture contains 20 mol.% of H2S, 70 mol.% of CO2 and 10 mol.% of water. It is supplied to a Claus 105

plant 8 via a line 7.

The absorbent freed of CO2 and H2S and becoming available from the regeneration column 5 via the line 16 is supplied to the absorption columns 2 and 13 via the lines 17 110 and 18, respectively.

EXAMPLE II

The reference numerals refer to Fig. 2. A gas stream of 10,000 Nm³/h, comprising 115 90% by volume of CO2, 7% by volume of H₂0, 2% by volume of hydrocarbons and 1% by volume of H₂S, is introduced between the eighth and ninth tray from the bottom of the absorption column 102 (which contains 18 trays). The pressure in the column is 1.05 bar abs. and the temperature 40°C. The gas becoming available from this column via the line 103 (9,790 Nm³/h) still contains 100 ppm by volume of HS and further 91% by volume of CO2, 7% by volume of H20 and 2% by volume of hydrocarbons. The loaded 7.0 mol.%, supplied via the line 1 to the absorbent in column 102, comprising a 24%

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104 into the 12-tray regeneration column 105. The regeneration column is heated with steam to a bottom temperature of 110°C, the pressure being 1.4 bar abs. From this column via the line 106 the desired gas becomes available at a rate of 2260 Nm³/h and having a composition of 47% by volume of H.S, 47% by volume of CO₂ and 6% by volume of H₂O. Of this gas, 210 Nm³/h is dis-10 charged via the line 107 to a Claus plant, and 2050 Nm³/h is introduced below the first tray from the bottom of absorption column 102 via the line 112. The gas velocity in this column is 1.5 m/sec. The absorbent regenerated in the regeneration column 105 is supplied via the line 117 to the absorption column 102 at a rate of 110 m³/h.

WHAT WE CLAIM IS:-

1. A method for increasing the H₂S/CO₂ 20 ratio in a gas mixture (hereinafter referred to as the starting gas), in order to prepare a gas mixture having an H2S/CO2 ratio suitable for use in a Claus process (hereinafter referred to as the desired gas), characterized 25 in that H₂S and CO₂ of the starting gas are absorbed in an aqueous solution of an amine (further to be called an amine liquid absorbent) in an absorption column, in that the absorbed gas is liberated from the resultant 30 loaded amine liquid absorbent in a regenerated column, in that a portion of the gas becoming available from the regeneration column is treated with amine liquid absorbent in an absorption column under such condi-35 tions that HaS is absorbed selectively relative to CO2 in the amine liquid absorbent and the loaded amine liquid absorbent obtained in this manner is introduced into the said regeneration column, and in that the magnitude of the said portion of the gas becoming available from the regeneration column is so selected that this gas is the desired gas, and the remainder of the gas becoming available from the regeneration col-45 umn (further referred to as the resultant gas) is removed.

2. A method as claimed in claim 1, characterized in that H-S from the starting gas is absorbed in the amine liquid absorbent

50 selectively relative to CO2.

3. A method as claimed in any one of claims 1-2, characterized in that the starting gas and the desired gas are treated in the same absorption column simultaneously 55 with the amine liquid absorbent.

4. A method as claimed in claim 3, characterized in that the starting gas and the desired gas are introduced into the absorption column at different points.

5. A method as claimed in claim 4, characterized in that the desired gas is introduced into the absorption column at a point below the point of introduction of the starting gas.

6. A method as claimed in any one of the preceding claims, characterized in that the absorption column in which the desired gas is treated with the said amine liquid absorbent contains few than 20 contact

trays.
7. A method as claimed in any one of characterized in that the preceding claims, characterized in that the gas velocity in the absorption column in which the desired gas is treated with the said

amine liquid absorbent, is at least 1m/sec.

8. A method as claimed in any one of the preceding claims, characterized in that the temperature of the amine liquid absorbent in the absorption column in which the desired gas is treated with the said amine liquid absorbent is from 15 to 50°C.

9. A method as claimed in any one of the preceding claims, characterized in that the desired gas is treated at approximately atmospheric pressure with the amine liquid

10. A method as claimed in any one of the preceding claims, characterized in that the amine liquid absorbent consists of an

aqueous solution of a polyalkanol amine.

11. A method as claimed in claim 10, characterized in that the polyalkanol amine is di-isopropanol amine.

12. A method as claimed in claim 10. characterized in that the polyalkanol amine is methyl disthanol amine.

13. A method as claimed in any one of the preceding claims characterized in that the aqueous solution of an amine also contains sulfolane.

14. A method as claimed in any one of the preceding claims, characterized in that the resultant gas mixture having a desired H₂S/CO₂ ratio is subjected to a Claus process.

15. A method as claimed in claim 1, substantially as described with special reference to the Examples.

> R. C. ROGERS Chartered Patent Agent, Shell Centre, London SE1 7NA Agent for the Applicants.

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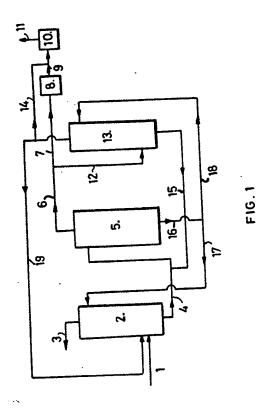
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1551692 COMPLETE SPECIFICATION

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1551692

COMPLETE SPECIFICATION

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Sheet 2

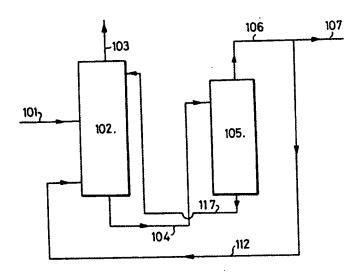


FIG. 2